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Influence of Drying Techniques on Some Properties of Nonfat Dried Milk

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Abstract

The bulk density, particle density, sinkability, and dispersibility of nonfat dried milk was varied by vacuum shelf drying, instantizing, and foam-spray-drying techniques. Good sinking and dispersing properties were obtained only by vacuum shelf drying, instantizing conventional spray-dried material and foam-spray-drying with low levels of CO_2 or N_2 incorporated into the concentrate prior to drying. All spray-dried powders having instant action were at least partially aggregated. The bulk density of these aggregated materials was relatively low. Incorporation of low levels of CO_2 into the concentrates was done by direct injection of the liquefied gas under pressure, decomposition of ammonium carbonate, and addition of CO_2 gas immediately ahead of the high pressure pump. High bulk density products having good sinking and dispersing properties were obtained by adding detergents to the concentrate and spray drying under conditions which produced in-dryer particle aggregation.

Improved control of the bulk density, particle size, and color of spray-dried coffee and tea by regulation of the amount and type of gas in the feed of a conventional spray dryer was claimed by Reich and Johnston in 1957 (18).

Slightly later, Coulter and Pyne (7, 17) demonstrated that a similar method could be used to improve the reconstitution characteristics of whole and skimmilk powders. They noted that the foamed nonfat dry milk (NDM), though superior to conventional spray-dried materials, did not reconstitute as easily as commercial powders instantized by rewetting, agglomerating, and redrying spray-dried particles. They also noted that the physical structure of foam-dried particles could be influenced by the type of gas used in the process, but believed this observation was not advantageous because of limitations of pilot plant equipment.

Equipment limitations were overcome by the invention of Hanrahan et al. (10). This novel method of injecting gas into liquids before spray drying had already been applied to the dehydration of wheys (12) and milks (2, 11), and had led us to study intensively the relationships between powder structure and properties. Our paper reports the results of such investigations as they relate to the improvement of the reconstitution properties of nonfat dried milk (NDM) by variation of drying technique. Also presented are additional methods to spray dry foamed milk concentrates without modifying conventional spray-drying equipment.

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Methods and Materials [1]

Skimmilk foams were dried under vacuum, using minor modifications of the method of Sinnamon et al. (20). Variation in bulk density of the powder was achieved by changing the amount of nitrogen mixed into the concentrate before drying, thus controlling the height of the puff.

Spray-dried foams of skimmilk were produced by high pressure gas injection using methods described by Hanrahan et al. (11). The 2.74-m Swenson dryer used was ordinarily equipped with a 1.02-mm spray nozzle and set to utilize 197.6 m^3 of 132 C air per min during drying. Feed rates to this dryer were determined by measuring the weight loss of the feed supply tank per min. A Corblin diaphragm pump (American Instrument Co., Silver Spring, Md.) was used to inject liquid CO_2 into the system when this material was used. A setup more adequately engineered to use liquefied gases with larger dryers has been described by Selman and Hanrahan (19).

Similar spray-dried foams were produced by bleeding small, measured quantities of CO_2 or N_2 gas into the concentrate immediately ahead of the high pressure pump. The gas from a high pressure tank was reduced to 4.1 atm and fed through a flow meter into the line connecting a small low pressure (3.4 atm) centrifugal pump to the high pressure pump used for atomization. A variable speed drive on the

[1] Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

high pressure pump allowed the maintenance of constant feed rate after gas incorporation.

The decomposition of carbonates was also used to produce CO_2 in the concentrates to develop foam structure during spray drying. This was done by mechanically metering solutions of carbonate under investigation into the milk concentrate immediately ahead of the high pressure pump.

Particles of NDM were instantized or agglomerated on laboratory scale by placing the powder sample in a chamber and reducing the pressure over the powder to 1 mm. Pressure in the chamber was then increased to 20 mm by rapid introduction of water vapor. Immediately, the wet aggregates were redried by again reducing the pressure over the powder bed to 1 mm and heating. The dried aggregates were broken through a 13-mesh screen before further analysis.

Bulk density of the powder was calculated from the measured volume occupied by a lightly tapped 10-g sample of milk powder.

Approximate particle density was measured with a variation of the Manus-Ashworth method, in which decalin was used as the displaced medium (5).

Sinkability of the powders was determined according to Bullock and Winder's method (6), and solubility index according to ADMI procedure (1).

Dispersibility of the powders was determined by the modification of Stone's procedure described by Kontson et al. (14).

Particle size distribution in the powders was determined by mechanically shaking a 150-g sample through a stack of U. S. Standard graded screens (Fisher Scientific Co., Silver Springs, Md.) and weighing the amount retained by each screen size. Moisture in the powders was measured by toluene codistillation, as described by Bidwell and Sterling (16).

All skimmilk used was made by separating fresh whole milk obtained from herds maintained at the Agricultural Research Center, Beltsville, Maryland.

Chemicals used were all reagent grade. Tween 60 and 80, Atmos 300, and Span 80, commercial detergents, were from the Atlas Chemical Industries, Inc., Chemical Division, Wilmington, Delaware. Samples of commercial instantized NDM were purchased at a local supermarket.

Results

Table 1 presents a résumé of the principal types of NDM studied and some of their physical properties. From this table it can be seen that both good sinking and dispersing properties were observed only with commercially instantized powder, vacuum shelf dried powder, conventionally spray-dried powder aggregated by wetting and redrying on laboratory scale, conventionally spray-dried powders containing surfactants, and foam-spray-dried powders made from concentrates containing low levels of CO_2.

From a closer examination of powders dried by foaming with small amounts of CO_2 and N_2, it was noted that high sinkability and dispersibility values were found together only with those powders that had undergone particle aggregation in the dryer. High moisture content in the powder tended to favor this aggregation. At low levels of CO_2 incorporation into the concentrate prior to drying, the degree of in-dryer aggregation and dispersibility characteristics of the powder could be varied by controlling production conditions, as illustrated in Table 2. Here the decomposition of NH_4HCO_3 was used to supply the gas.

Microscopic examination of the various particle size fractions listed in Table 2 showed that particle aggregation was responsible for increased sinkability. Aggregation usually increased particle size; however, at higher spray pressure particle size decreased. This resulted in a decrease in the size of the aggregates formed during drying.

During this work, some question arose as to the relationship between the solubility of the gas in the concentrate and the dispersibility of the powders. Since our high pressure gas injection apparatus would not permit the accurate metering of small quantities of N_2 into the concentrate, a comparative study was made by mixing small quantities of the gases under consideration into the concentrate, at low pressure, directly ahead of the high pressure pump. Results obtained are presented in Table 3.

Discussion

When drying skimmilk with a conventional pressure atomizing spray dryer, the operator must select methodology which gives a product most suitable for the projected end use, since it is apparent that, as yet, no product can be made from unmodified skimmilk which will have high bulk density, excellent sinking properties, and complete dispersibility.

The general idea, presented by Harper et al. (13), that good dispersibility would not be found in high bulk density dried milk products, was further confirmed by the observations reported. It may be that the protein destabilizing effect of high concentrations of phosphate reported by Fox et al. (8) is operative during the reconsti-

TABLE 1

Effect of drying technique on properties of skimmilk powder

Sample no.	Drying technique employed	Powder properties: Bulk dens. (g/cc)	Part. dens. (g/cc)	Sink. (%)	Disp. (%)
1	Commercial instantized	.34	1.34	98	97
2	Vacuum shelf dried—high puff	.17	1.35	95	100
3	Vacuum shelf dried—low puff	.29	1.41	91	98
4	Conventional spray-dried[a]	.65	1.20	82	82
5	Conventional spray-dried—lab scale steam aggregated	.55	1.21	95	98
6	Foam-spray-dried-N_2 injection 1.87 liters/kg conc	.40	.80	44	100
7	Foam-spray-dried plus steam aggregation, N_2 injection 1.87 liters/kg conc	.38	.90	56	100
8	Foam-spray-dried plus .1% Tween 80 in conc N_2 injection 1.87 liters/kg conc	.39	.81	43	100
9	Conventional spray-dried plus .1% Tween 80 in conc	.67	1.20	98	97
10	Conventional spray-dried plus .1% Tween 60 in conc	.64	1.21	97	96
11	Conventional spray-dried plus .1% Atmos 300 in conc	.67	1.15	98	97
12	Conventional spray-dried plus .1% Span 80 in conc	.66	1.18	97	93
13	Foam-spray-dried-liquid CO_2[b] injection, 1.24 liters/kg conc	.50	.92	89	99
14	Foam-spray-dried-liquid CO_2 injection, 5.04 liters/kg conc	.20	.98	43	99
15	Foam-spray-dried-liquid CO_2 injection, 12.82 liters/kg conc	.15	.91	32	97
16	Foam-spray-dried NH_4HCO_3 in feed equivalent to .12 liters CO_2/kg conc[c]	.62	1.07	99	91
17	Foam-spray-dried NH_4HCO_3 in feed equivalent to .25 liters CO_2/kg conc[c]	.48	.95	96	98
18	Foam-spray-dried NH_4HCO_3 in feed equivalent to .56 liters CO_2/kg conc[c]	.43	.92	86	99

[a] All spray drying in this table done by using 42-45% T.S. in feed, 1.02-mm nozzle, 132 C drying air temperature, and atomizing pressure of 102 atm.

[b] All CO_2 expressed in terms of standard volume of gas in concentrates.

[c] Calculation based on volume CO_2 (expressed in terms of standard temperature and pressure) released on decomposition of bicarbonate. Volume of NH_3 liberated is not included in the figure.

tution of high bulk density products and can lead to a decrease in their dispersibility.

The bulk density of spray-dried NDM can easily be decreased by use of foam spray drying to the point where high dispersibility values are obtained. However, as the bulk density of the powder decreases because of particle density decrease due to gas entrapment, a point is reached where the particles tend to float on the surface of water and sinkability is drastically reduced. Foam-spray-dried NDM products produced by injecting relatively large amounts of gas all float. Those foam-spray-dried products that sink like instantized conventional spray-dried products are all partially aggregated. Since it was noted that the sinking properties of those powders were lost on mechanically breaking up the aggregates by grinding, it is believed that foam-spray-dried NDM particles containing small amounts of gas must also be aggregated to have good sinking properties.

Our dryer apparently produces sufficient aggregation in its dehydrating chamber to produce instant type powders when low levels of gas are incorporated into the feed and drying conditions favoring the production of high moisture powders are used. By observing the drying operation through portholes cut into the dryer wall, we noted that some of the powder was

TABLE 2

Skimmilk concentrates foam-spray-dried with NH_4HCO_3 incorporation at a rate equivalent to 1.41 liter CO_2/min[a]

	Varied % T.S.			Varied conc temp			Varied spray pressure		
Concentrate									
% T.S.	40	45	50	45	45	45	45	45	45
C	38	38	38	60	38	16	38	38	38
Atmospheres	102	102	102	102	102	95	85	119	153
Kg/min	3.63	4.22	4.85	3.86	4.13	4.54	3.72	4.45	5.08
Dry milk									
% H_2O	2.4	3.4	6.2	4.0	5.3	6.5	4.0	4.0	4.0
Bulk dens.	.43	.45	.42	.43	.45	.45	.43	.48	.55
Particle dens.	.87	.93	1.00	.85	.91	.97	.89	.87	.87
% Disp.	97	97	96	100	100	99	97	98	99
% Sink.	45	80	98	48	94	94	61	82	87
Particle size distr. wt %									
$<105\ \mu$	12	10	8	8	14	13	19	27	41
105-250 μ	75	50	15	61	44	22	31	40	38
250-500 μ	12	32	29	29	34	24	32	27	19
500-710 μ		6	19	2	8	15	10	4	2
$>710\ \mu$		2	29		1	26	7	1	

[a] Calculation based on volume CO_2 (expressed in terms of standard temperature and pressure) released on decomposition of bicarbonate. Volume of NH_3 liberated is not included in the figure.

TABLE 3

Skimmilk concentrate of 45% T.S. foam-spray-dried at 34 C (nozzle 1.02 mm, dryer temperature 132 C) with small amounts of CO_2 or N^2 injected ahead of the high pressure pump

	Foamed with CO_2			Foamed with N_2		
Concentrate						
Liters gas/min	1.41	2.82	4.23	1.98	3.95	4.80
Atm	102	102	102	102	102	88
Kg/min	4.08	4.08	4.17	4.31	4.31	4.26
Dry Milk						
% H_2O	4.2	2.6	2.5	4.0	3.6	3.8
Bulk dens.	.39	.28	.24	.41	.34	.32
% Disp.	99	98	99	99	99	99
% Sink.	72	39	24	87	44	48
Solubility index	.10	.10	.10	.07	.07	.05

blown against the upper sidewall of the dryer cone and tended to stick there until removed by the chain sweeps whenever in-dryer aggregation occurred. We therefore believe that agglomerated foam-spray-dried NDM will be produced only in dryers whose air flow patterns allow a large number of collisions of partially dried particles before drying is completed. Good sinking properties would not be expected from foam-spray-dried products made in dryers having parallel air and product flow patterns.

Also evident is the improvement in sinkability of the foam-spray-dried NDM by decreasing gas content of the powder particle, resulting in decreased dryer efficiency. Therefore, when using low levels of CO_2 or N_2 in the dryer feed, the economic gains resulting from drying high solids concentrate, as cited by Bell et al. (2) are reduced.

While the apparatus necessary to convert an industrial type dryer to foam-spray drying with CO_2, as described by Selman et al. (19), is relatively simple, similar results are obtainable by metering ammonium bicarbonate into the concentrate immediately ahead of the high pressure pump. The pH of milk and the temperatures it encounters during drying will decompose the carbonate, releasing CO_2 and ammonia. Theoretically, if any ammonia is retained in the powder, it should replace that originally present in the milk and lost during processing, thereby leading to a more fresh flavored product. However, in our limited experience, it is not possible to distinguish between powders foamed by liquid CO_2 injection or bicarbonate decomposition on the basis of taste or any physical property.

No problems were encountered in pumping

the carbonate containing concentrates as described. Increase in pump speed was not necessary to maintain atomization pressure when carbonate solutions were metered into the concentrate. It is possible that chemical decomposition resulting in release of CO_2 occurs after passage through the high pressure pump and, therefore, would not interfere with its operation.

It is obvious that the carbonate added to milk for the purpose of CO_2 production must be considered an additive, and subject to those legal restrictions now in effect.

When powders are foam-spray-dried by injecting relatively large amounts of either CO_2 or N_2 in the powders, the observed structures of the particles are grossly different. The coarse foam structure of the soluble gas containing particles as opposed to the fine grained foam structures produced by nitrogen injection has already been described by Pyne (17). The effect of this structural variation on the porosity, density, and surface areas of these powders has been studied by Berlin et al. (3, 4) and found to be significant. However, as this study shows, when relatively small amounts of either CO_2 or N_2 are incorporated into the concentrate before atomization, the resultant powders are much alike as far as their dispersibility and sinkability are concerned.

Caution must be exercised in those experiments when either N_2 or CO_2 is incorporated into the concentrate immediately ahead of the pump, as described in this paper. Our own experience showed that this could be done without apparent trouble only when one to three liters of gas (at 1-atm pressure) were injected into the system per min. In this case, speeding up the pump allowed maintenance of proper atomization pressure. When larger amounts of gas were used, not only did sinkability of the product decrease but difficulty was encountered in maintaining a uniform atomization pressure.

The practice of introducing gas ahead of the pump has been discouraged by a leading high pressure pump manufacturer, but in view of the dearth of information on the solubility of gases in milk concentrates under pressure, more experience must be gathered in this area before the incorporation of small quantities of gas directly ahead of the pump, for the purpose of foam drying, can be shown to be commercially unfeasible.

Data in Table 2 show that when either the solids in the concentrate being dried are increased or its temperature is dropped (both conditions favoring an increase in viscosity), the speed of the pump had to be increased to maintain constant atomizing pressure. Although there is little theory developed to predicate the effect of viscosity change on atomization phenomena, it is obvious from our results that the energy required for atomization increases as the viscosity increases. A simple formula that can be used to calculate the energy requirements for atomization using pressure nozzles has been given by Marshall (15) as:

$$E_n = 19.2\ QP$$

where E_n = energy used ft-lb/min.
Q = flow rate through nozzle gal/min.
P = total pressure drop lb/sq in.

Our data can be converted to a form suitable for use with this equation, using the specific gravity data and temperature coefficients already in the literature (9).

When the atomization pressure was varied and all other factors held constant, the number of large aggregates in the powder decreased as atomization pressure increased. However, aggregation still occurred in powders produced with high atomization pressure, and they maintained good sinking and dispersing characteristics.

Since only three of the variables listed in Table 2 could be studied per day, those slight variations in moisture content in powders produced by nearly comparable drying conditions are attributed to daily variations in humidity of of the air entering the dryer.

The best bulk density, dispersibility, and sinkability values in our study were obtained when concentrates containing detergents were spray dried in conventional fashion. Although the powders were partially aggregated, it is obvious that the additives also were effective in maintaining protein solubility during the reconstitution process. It is possible that more suitable additives can be found, and it is along these lines that some of our present research is proceeding.

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